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SYNTHESIS CHARACTERIZATION AND CRYSTAL AND MOLECULAR
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BUFFALO DEPT OF CHEMISTRY R B HALLOCK ET AL. 10 OCT 84 1/1

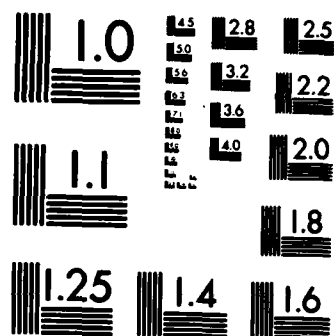
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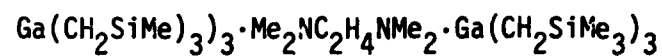
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Synthesis, Characterization and Crystal and Molecular Structure of



by

R. B. Hallock, W. E. Hunter, Jerry L. Atwood and

O. T. Beachley, Jr.

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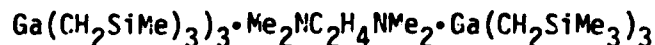
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Synthesis, Characterization and Crystal and Molecular Structure of



by

R. B. Hallock^{1a}, W. E. Hunter^{1b}, Jerry L. Atwood^{1b} and
O. T. Beachley, Jr.*^{1a}

Abstract

The complex $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$, prepared from $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$, has been fully characterized by analysis, standard spectroscopic methods, cryoscopic molecular weight measurements and an X-ray structural study. The compound crystallizes in the monoclinic space group $P2_1/n$ with unit cell dimensions $a = 9.911(4)\text{\AA}$, $b = 16.427(6)\text{\AA}$, $c = 14.770(6)\text{\AA}$, $\beta = 90.43(4)^\circ$ and $Z = 2$ for $d_{\text{calcd}} = 1.08 \text{ g cm}^{-3}$. Full-matrix least-squares refinement led to a final R value of 0.075 for 1598 observed reflections. The molecule lies on a crystallographic inversion center. The geometry about the gallium atom is distorted tetrahedral with a Ga-C(av) distance of $1.99[1]\text{\AA}$ and a Ga-N distance of $2.241(9)\text{\AA}$.

Introduction

The trimethylsilylmethyl group is considered to be a bulky substituent with larger steric effects than those of a methyl group. Consequently, the trimethylsilylmethyl Group 3 derivatives $M(\text{CH}_2\text{SiMe}_3)_3$ ($M=\text{Al}, \text{Ga}, \text{In}$) are considered to be weaker Lewis acids than the corresponding methyl derivatives. For example, diethylether can be readily removed from the gallium² and indium³ derivatives but the strong base NMe_3 forms stable complexes with all of these Group 3 compounds. Diamines of the type $\text{Me}_2\text{N}(\text{CH}_2)_n\text{NMe}_2$ ($n=2,3$) react with MMe_3 ($M=\text{Al}, \text{Ga}, \text{In}$) to form 1:2 complexes⁴ $\text{Me}_3\text{M} \cdot \text{Me}_2\text{N}(\text{CH}_2)_n\text{NMe}_2 \cdot \text{MMe}_3$. Cryoscopic molecular weight measurements of these methyl complexes in benzene solution are consistent with monomeric behavior and little dissociation into 1:1 complexes and free Lewis acid. During the course of some of our investigations of the chemistry of $\text{M}(\text{CH}_2\text{SiMe}_3)_3$, $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ was reacted with a large excess of $\text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$. The isolated product $(\text{Me}_3\text{SiCH}_2)_3\text{Ga} \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ was fully characterized by analysis, standard spectroscopic methods, cryoscopic molecular weight measurements and an X-ray structural study. This structural study represents only the second report of a structure of a neutral trialkylgallium-amine donor-acceptor complex and the first with an alkyl group other than methyl.

Experimental Section

The organogallium compounds described in this investigation were oxygen and moisture sensitive and were manipulated in a vacuum line or a purified argon atmosphere. The reagent $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ was synthesized by the literature procedure.² The ligand $\text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$ (TMED) was dried over P_2O_5 and was vacuum distilled immediately prior to reaction. Infrared spectra were recorded in the range $4000\text{--}250\text{ cm}^{-1}$ by means of a Perkin-Elmer 683 spectrometer. The spectra were recorded as Nujol mulls by using CsI plates. Bands due to mulling agents have been omitted. The ^1H NMR spectrum was recorded at 90 MHz and ambient temperature by using a Varian EM-390 spectrometer. Chemical shift data are reported in δ units (ppm) and are referenced to SiMe_4 as 0.00 and C_6H_6 as 7.13. Molecular weight measurements were obtained cryoscopically in benzene by using an instrument similar to that described by Shriver.⁵

Synthesis of $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$.

The title compound was prepared by vacuum distilling a large (ten fold) excess of $\text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$ onto 0.785 g (2.37 mmol) $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ in a reaction tube equipped with a Teflon valve. After warming to room temperature, the reaction mixture was stirred for 5 h. The excess ligand was removed by vacuum distillation at room temperature to leave a viscous yellow liquid. This liquid was then heated to 45° under high vacuum and colorless crystals sublimed to the cooler portions of the tube. The crystals were isolated and resublimed at 45° . The final product $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ (0.71 g, 0.912 mmol, 77.3%) was isolated as colorless crystals: mp $89\text{--}91^\circ\text{C}$, ^1H NMR (benzene, δ) + 2.34 (s, 1.0, $\text{CH}_2\text{-TMED}$), + 1.88 (s, 2.9, Me-TMED), + 0.20 (s, 11.4,

Me-CH₂SiMe₃), - 0.30 (s, 3.0, CH₂-CH₂SiMe₃); IR(Nujol, cm⁻¹) 1288 (w), 1252 (m), 1240 (s), 1162 (vw), 1128 (w), 1095 (vw), 1012 (m,sh), 999 (m), 982 (s), 960 (m), 945 (m), 860 (s,sh), 850 (s), 822 (vs), 783 (w), 748 (m), 721 (m), 677 (m), 610 (vw), 562 (w), 538 (w), 513 (m), 459 (vw); cryoscopic molecular weight; formula weight Ga(CH₂SiMe₃)₃·Me₂NC₂H₄NMe₂·Ga(CH₂SiMe₃)₃, 778.9. Molality, obsd mol wt:0.0990, 549; 0.0687, 491; 0.0540, 452. Hydrolysis: 0.0900 g (0.116 mmol) hydrolyzed in 6 M HCl for 48 h at 100°C gave 0.697 mmol SiMe₄, calcd 0.696. (SiMe₄ from hydrolysis was purified by fractionation through two -78°C traps and isolated at -196° and the quantity was determined by PVT measurements on the vacuum line.)

Collection of X-Ray Diffraction Data and Solution of Structure for Ga(CH₂SiMe₃)₃·Me₂NC₂H₄NMe₂·Ga(CH₂SiMe₃)₃.

Single crystals of the air-sensitive compound were isolated by sublimation at 65°C from an attempted reaction between Ga(CH₂SiMe₃)₃ and LiH in Me₂NC₂H₄NMe₂ and were sealed under N₂ in thin-walled glass capillaries. Final lattice parameters as determined from a least-squares refinement of ((sin θ)/λ)² values for 15 reflections (θ > 20°) accurately centered on the diffractometer are given in Table I.

Data were collected on an Enraf-Nonius CAD-4 diffractometer by the θ-2θ scan technique. This method has been previously described.⁶ A summary of data collection parameters is given in Table I. The intensities were corrected for Lorentz and polarization effects.

Calculations were carried out with the SHELX system of computer programs.⁷ Neutral-atom scattering factors for Ga were taken from Cromer and Waber.⁸ Scattering factors stored within the SHELX program were used

for the other atoms. The scattering factor for Ga was corrected for both the real and imaginary components of anomalous dispersion using the table of Cromer and Liberman.⁹ The position of the Ga atom was located using the MULTAN80 system of programs.¹⁰ Difference Fourier maps phased by this atom readily revealed the positions of the remaining nonhydrogen atoms. Full-matrix least-squares refinement with isotropic thermal parameters led to $R = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.090$. Further refinement with anisotropic thermal parameters led to $R = 0.080$. At this point a difference Fourier map was computed and the hydrogen atoms were located. Further refinement led to final values of $R = 0.075$ and $R_w = 0.075$.

A final difference Fourier showed no feature greater than $0.6 \text{ e}/\text{\AA}^3$. The weighting scheme was based on unit weights; no systematic variation of $w(|F_o| - |F_c|)^2$ vs. $|F_o|$ or $(\sin \theta)/\lambda$ was noted. The final values of the positional parameters of atoms other than hydrogen are given in Table II. Positional parameters for all atoms (Table IV-S) and anisotropic thermal parameters (Table V-S) are deposited as Supplementary Material.

Results and Discussion

The complex $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ has been isolated from a reaction mixture containing $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ and a large excess of $\text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$ (TMED). It is of interest that the product has both basic nitrogen atoms complexed with Lewis acids, even though the preparative reaction used an excess of TMED. Cryoscopic molecular weight measurements in benzene solution and the relatively low sublimation temperature (45-65°C) for such a large and heavy molecule suggest that the complex partially dissociates both in solution and in the vapor phase. The observed molecular weight is significantly lower than the formula weight of the compound for all solutions studied. Furthermore, the observed molecular weight is concentration dependent, decreasing with concentration. In contrast, the related complexes of the methyl metal derivatives⁴ $\text{Me}_3\text{M} \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{MMe}_3$ (M = Al, Ga, In), prepared from a reaction mixture having one mole TMED and two moles MMe_3 , do not exhibit any solution properties suggestive of significant dissociation. It is also noteworthy that the melting point of $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$ (89-91°C) is lower than that of $(\text{GaMe}_3)_2 \cdot \text{TMED}$ ⁴ (102-103°C), which is as expected if there is partial dissociation of the former complex upon heating. The occurrence of dissociative equilibria during the isolation of the complex can also be used to account for the formation of $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$ rather than $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{TMED}$, especially from a reaction mixture using excess TMED. If

$\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{TMED}$ is the initial product, a dissociative reaction



would lead to the formation of $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ and TMED. Since free TMED is the most volatile component, TMED would be removed by vacuum distillation during isolation and/or sublimation to leave $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$, as observed.

The X-ray structural study demonstrates that the crystal consists of isolated molecules of $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$, separated by normal van der Waals distances. There are no abnormally short intermolecular contacts. Selected interatomic distances are presented in Table III. Figure 1 shows the scheme used in labeling the atoms, while Figure 2 provides a stereoscopic view of the unit cell. The molecule lies on a crystallographic inversion center. The gallium and nitrogen atoms are in rather distorted tetrahedral environments. The gallium-alkyl distances ($\text{Ga}-\text{CH}_2\text{SiMe}_3$) are $\text{Ga}-\text{C}(1) = 2.00(1)\text{\AA}$, $\text{Ga}-\text{C}(5) = 1.99(1)\text{\AA}$ and $\text{Ga}-\text{C}(9) = 1.98(1)\text{\AA}$, the average $\text{Ga}-\text{C}(\text{sp}^3)$ distance is $1.99[1]\text{\AA}$.¹¹ These distances are shorter than the gallium-alkyl distances¹² in $\text{KGa}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ which range from $2.030(9)$ to $2.003(9)\text{\AA}$, the average value being $2.029[14]\text{\AA}$. The gallium-nitrogen distance in $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$ is $2.241(9)\text{\AA}$ which is significantly longer than that observed in the urotropine-trimethylgallium adducts,¹³ $\text{GaMe}_3 \cdot \text{C}_6\text{H}_{12}\text{N}_4$ and $(\text{GaMe}_3)_2 \cdot \text{C}_6\text{H}_{12}\text{N}_4$, $2.139(17)$ and $2.138(9)\text{\AA}$, respectively. The average $\text{Ga}-\text{C}$ bond distances in the urotropine complexes are $1.992[14]$ and $1.981[19]\text{\AA}$, respectively. For comparison, an electron diffraction study¹⁴ of

$\text{Me}_3\text{Ga}\cdot\text{NMe}_3$ reveals a Ga-N distance of 2.20(3)Å and an average Ga-C distance of 1.998[4]Å. The angles around gallium in $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2\cdot\text{TMED}$ in decreasing order are $\text{C}(1)\text{-Ga-C}(9) = 116.9(5)^\circ$, $\text{C}(1)\text{-Ga-C}(5) = 116.6(4)^\circ$, $\text{C}(5)\text{-Ga-C}(9) = 116.2(5)^\circ$, $\text{C}(1)\text{-Ga-N}(1) = 102.1(4)^\circ$, $\text{C}(9)\text{-Ga-N}(1) = 101.0(4)^\circ$ and $\text{C}(5)\text{-Ga-N}(1) = 99.3(4)^\circ$. The Ga-C(α)-Si angles which range from 125.9(6)° to 121.1(6)° are substantially greater than the ideal tetrahedral angle of 109.47°. All other parameters of the molecule seem normal.

Acknowledgment. This work was supported in part by the Office of Naval Research (O.T.B.) and the National Science Foundation (Grant CHE-81-11137 (J.L.A.)).

Supplementary Material Available: Tables IV-S, V-S, VI-S, showing positional parameters, anisotropic thermal parameters observed and calculated structure factor amplitudes, (pages). Ordering information is given on any current masthead page.

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$$[\sigma] = \left[\sum_{i=1}^N (\bar{d} - d_i)^2 / (N-1) \right]^{1/2}$$
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Table 1. Crystal Data for $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$

comp	$\text{Ga}_2\text{Si}_6\text{N}_2\text{C}_{30}\text{H}_{82}$
mol wt	778.90
space group	$\text{P}2_1/\text{n}$
cell constants	
a, Å	9.911(4)
b, Å	16.427(6)
c, Å	14.770(6)
β , deg	90.43(4)
cell vol, Å ³	2404.6
molecules/unit cell (Z)	2
ρ (calcd) g cm ⁻³	1.08
μ (calcd), cm ⁻¹	13.46
radiation	MoK α
max crystal dims, mm	0.30 x 0.20 x 0.15
scan width, deg	0.8 + 0.2 tan θ
std reflctns	040, 004
decay of stds	< 3%
reflctns measd	2519
2 θ range	2 - 40
obsd reflctns	1598
no. of parameters varied	181
GOF	2.99
R	0.075
Rw	0.075

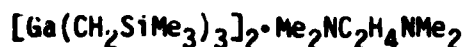
Table II

Final Fractional Coordinates for $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$

Atom	x/a	y/b	z/c	U(eqv)
Ga(1)	0.5077(1)	0.1498(9)	0.3729(6)	0.050
N(1)	0.9779(1)	0.0103(6)	0.7067(8)	0.051
C(1)	0.9457(2)	0.0336(7)	0.6391(2)	0.061
C(2)	0.9755(2)	0.0388(7)	0.6111(2)	0.061
C(3)	0.9853(2)	0.0240(7)	0.5701(2)	0.061
C(4)	0.9637(2)	0.0391(7)	0.5977(2)	0.061
C(5)	0.9357(2)	0.0491(8)	0.5666(2)	0.061
Si(2)	0.5673(5)	0.2064(3)	0.3913(3)	0.061
C(6)	0.5356(7)	0.2123(8)	0.3221(3)	0.061
C(7)	0.5356(7)	0.2123(8)	0.3221(3)	0.061
C(8)	0.5356(7)	0.2123(8)	0.3221(3)	0.061
C(9)	0.5356(7)	0.2123(8)	0.3221(3)	0.061
Si(1)	0.4911(5)	0.1471(3)	0.2133(3)	0.061
C(10)	0.4633(7)	0.1530(8)	0.1443(3)	0.061
C(11)	0.4633(7)	0.1530(8)	0.1443(3)	0.061
C(12)	0.4633(7)	0.1530(8)	0.1443(3)	0.061
C(13)	0.4633(7)	0.1530(8)	0.1443(3)	0.061
C(14)	0.4633(7)	0.1530(8)	0.1443(3)	0.061
C(15)	0.4633(7)	0.1530(8)	0.1443(3)	0.061

Table III

Bond Lengths (Å) and Bond Angles (deg) for



Atoms		Distance	Atoms		Distance
Ga(1)	-- C(1)	2.00(1)	Ga(1)	-- C(5)	1.99(1)
Ga(1)	-- C(9)	1.98(1)	Ga(1)	-- N(1)	2.241(9)
Si(1)	-- C(1)	1.87(1)	Si(1)	-- C(2)	1.86(1)
Si(1)	-- C(3)	1.86(1)	Si(1)	-- C(4)	1.85(1)
Si(2)	-- C(5)	1.83(1)	Si(2)	-- C(6)	1.85(1)
Si(2)	-- C(7)	1.85(2)	Si(2)	-- C(8)	1.81(2)
Si(3)	-- C(9)	1.85(1)	Si(3)	-- C(10)	1.87(2)
Si(3)	-- C(11)	1.85(1)	Si(3)	-- C(12)	1.86(1)
C(13)	-- C(13)	1.47(3)	C(13)	-- N(1)	1.51(2)
C(14)	-- N(1)	1.52(2)	C(15)	-- N(1)	1.50(2)
Atoms		Angle	Atoms		Angle
C(1)	-- Ga(1) -- C(5)	116.6(4)	C(1)	-- Ga(1) -- C(9)	116.9(5)
C(5)	-- Ga(1) -- C(9)	116.2(5)	C(1)	-- Ga(1) -- N(1)	102.1(4)
C(5)	-- Ga(1) -- N(1)	99.3(4)	C(9)	-- Ga(1) -- N(1)	101.0(4)
C(1)	-- Si(1) -- C(2)	112.1(6)	C(1)	-- Si(1) -- C(3)	110.2(6)
C(2)	-- Si(1) -- C(3)	105.9(7)	C(1)	-- Si(1) -- C(4)	112.9(6)
C(2)	-- Si(1) -- C(4)	103.0(7)	C(3)	-- Si(1) -- C(4)	107.3(7)
C(5)	-- Si(2) -- C(6)	111.1(7)	C(5)	-- Si(2) -- C(7)	113.4(8)
C(6)	-- Si(2) -- C(7)	105.1(9)	C(5)	-- Si(2) -- C(8)	109.2(8)
C(6)	-- Si(2) -- C(8)	109.8(9)	C(7)	-- Si(2) -- C(8)	105(1)
C(9)	-- Si(3) -- C(10)	110.3(6)	C(9)	-- Si(3) -- C(11)	112.2(6)
C(10)	-- Si(3) -- C(11)	103.3(7)	C(9)	-- Si(3) -- C(12)	111.2(7)
C(10)	-- Si(3) -- C(12)	100.4(8)	C(11)	-- Si(3) -- C(12)	108.2(7)
Ga(1)	-- C(1) -- Si(1)	121.1(6)	Ga(1)	-- C(5) -- Si(2)	125.9(6)
Ga(1)	-- C(9) -- Si(3)	122.5(6)	Ga(1)	-- N(1) -- C(13)	115.9(7)
Ga(1)	-- N(1) -- C(14)	108.2(7)	C(13)	-- N(1) -- C(14)	112(1)
Ga(1)	-- N(1) -- C(15)	106.6(7)	C(13)	-- N(1) -- C(15)	105(1)
C(14)	-- N(1) -- C(15)	109(1)			

Figures

Figure 1 Labeling of atoms in $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_3 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$.
(Trimethylsilyl groups shown as point atoms for clarity; Ortep-II diagram showing 30% probability contours of the thermal vibration ellipsoids of non-hydrogen atoms.)

Figure 2 Stereoscopic view of unit cell of $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$

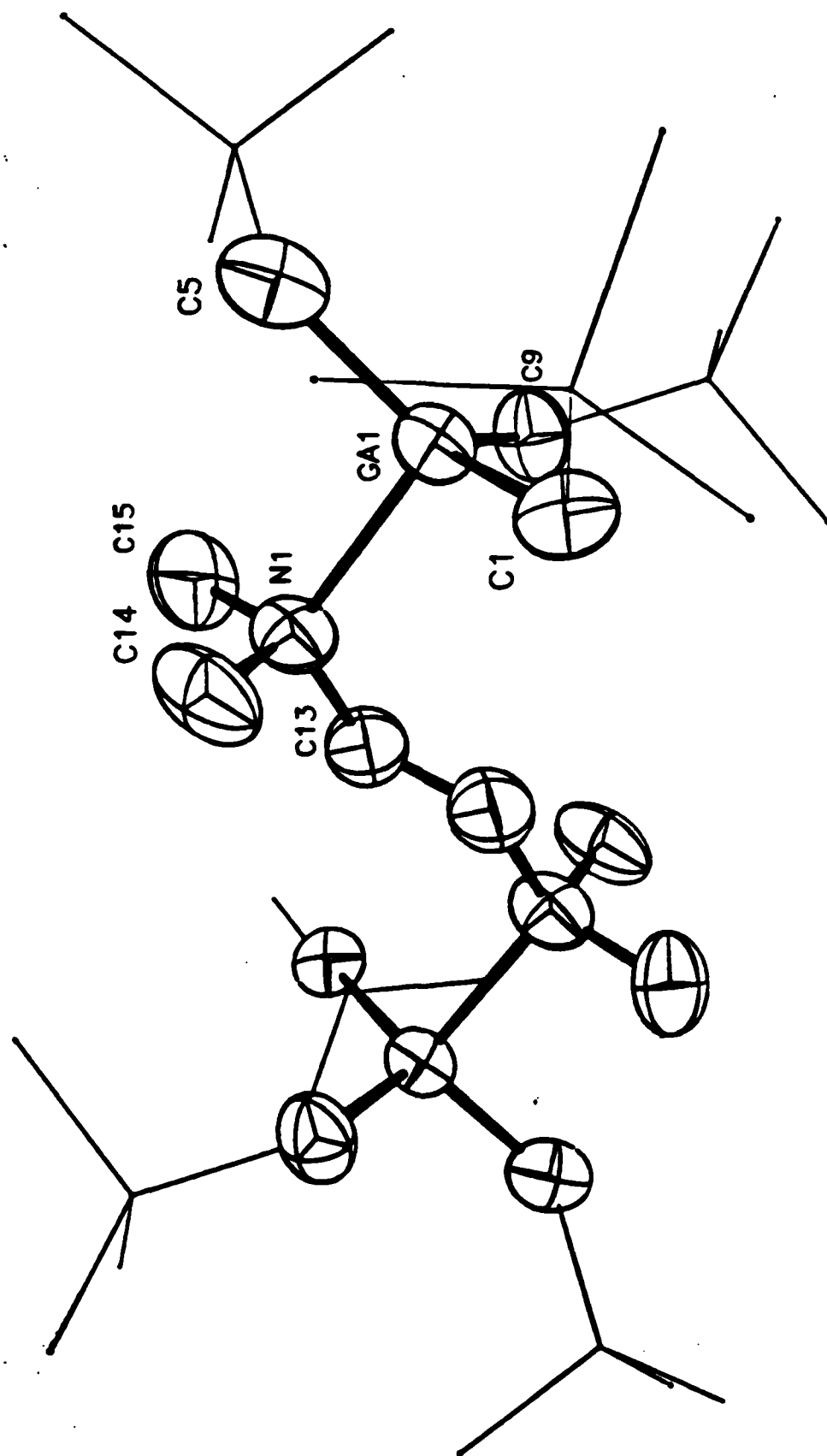


Figure 1

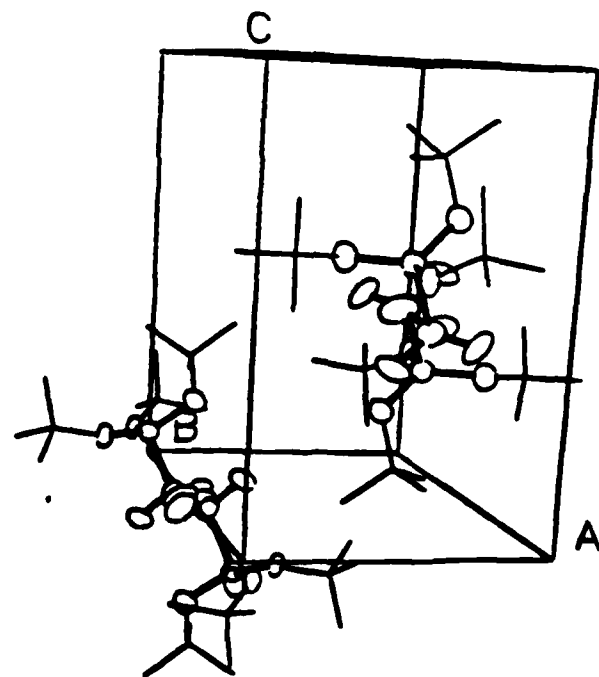
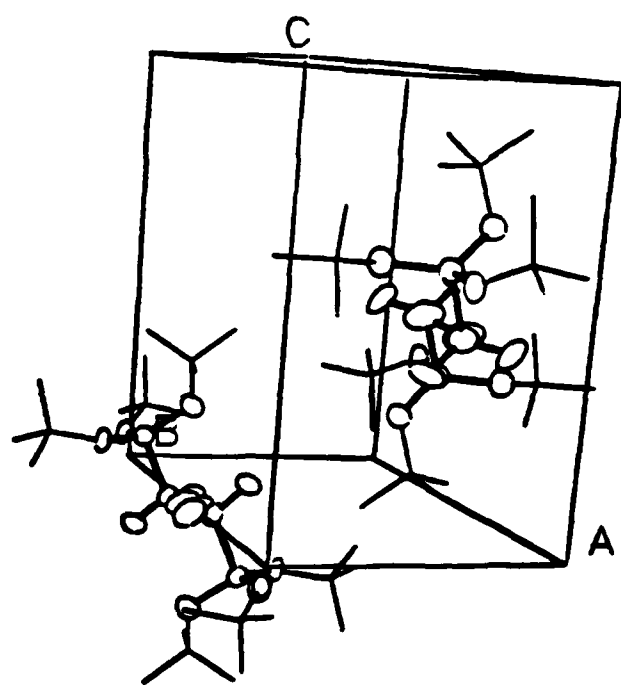


Figure 2

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